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Investigation into the improvement of the Small-scale Cook-off Bomb (SCB)

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A-1

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Titel	: Investigation into the improvement of the Small-scale Cook-off Bomb (SCB)
Auteur	: J.H.G. Scholtes, B.J. van der Meer
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Het TNO Prins Maurits Laboratorium is begin 1991 gestart met de constructie van de Small scale Cook-off Bomb test (SCB) zoals deze in de UN-manual, testserie 1 en 2, beschreven staat. De test is ontwikkeld in het kader van het onderzoek “Thermische Initiatie”. Met de SCB-test bepaalt men de responsie van een energetische stof, in opgesloten toestand, onderworpen aan een externe thermische belasting. Naast classificatie van een energetische stof, helpt deze test bij het vergroten van het inzicht in de responsie van energetische stoffen bij thermisch belasting, ook wel Cook-off genoemd.

Na een aantal inleidende testen met TNT, RDX en AN bleken parameters als netspanning, positie van verwarmingselementen en thermokoppels (TK's) een grote invloed op de temperatuurmeting te hebben. Deze invloeden zijn grondig onderzocht.

Het blijkt dat een netspanningsafwijking van 10 Volt, bij een referentiespanning van 220 Volt, kan leiden tot een relatieve temperatuursverandering van 9%. De combinatie van de TK-locatie en de locatie van de verwarmingselementen kunnen leiden tot temperatuursverschillen van meer dan 20% en kunnen daarmee de opwarmsnelheid van het experiment beïnvloeden. Daar de omwarmsnelheid bepalend kan zijn voor de responsie van een energetische stof, kan een gemeten verschil in de temperatuur dus het testresultaat beïnvloeden.

Verder is gebleken dat de beschreven opwarmsnelheid in de UN-manual niet gebruikt kon zijn in de gegeven resultaten. Ook was niet geheel duidelijk of de testen uitgevoerd werden met een constante opwarmsnelheid dan wel met een constante spanning.

Uit twee series van testen met energetische stoffen in opgesloten en niet-opgesloten toestand blijkt dat het uitvoeren van de testen in opgesloten toestand beter een reproduceerbaard is dan in niet-opgesloten toestand.

De SCB-test, in zijn huidige vorm, is samen met enkele andere Cook-off testen gepresenteerd op de “NIMIC Workshop on Cook-off” van juni 1993. Uitgaande van de UN-SCB-test wordt aanbevolen de test op de volgende wijze uit te voeren:

- voor de opwarming van het vat, drie in plaats van twee bandverwarmingselementen gebruiken om zo de temperatuur-gradiënten te verkleinen;
- het thermokoppel dat teruggekoppeld is naar de temperatuurscontroller niet inwendig maar uitwendig tussen de klemmen van de middelste bandverwarming te plaatsen zodat ook persbare energetische stoffen getest kunnen worden;

- de testen uit te voeren met een gecontroleerde constante opwarmsneldheid van 3 °C/minuut;
- de testen zo uit te voeren dat de energetische stof volledig opgesloten is.

De opwarmsnelheid van de SCB-test is volledig instelbaar en kan opwarmsnelheden bereiken zoals die bij de fuel fire test optreden. Door vervanging van testen zoals deze fuel fire test door de SCB-test zou de lucht en grondwatervervuiling sterk verminderd worden.

Naast het voorkomen en verzachten van de responsie van het Cook-off fenomeen en de bepaling van de typen bedreiging welke een Cook-off tot gevolg kunnen hebben, is de bepaling van de heftigheid van de response van groot belang. Een energetische stof, onderworpen aan een thermische belasting, die detoneert bij een temperatuur van 250 °C is gevaarlijker dan een stof die bij reeds 150 °C een milde verbranding vertoont. Daarom is de constructie en ontwikkeling van goed geïnstrumenteerde testen om de responsie van een energetische stof, onderworpen aan thermische belasting, te bepalen, essentieel voor het onderzoek van het Cook-off fenomeen.

CONTENTS

1 INTRODUCTION

The Small-scale Cook-off Bomb (SCB) test facility was established at TNO-PML in 1991. The SCB test is described in the UN manual "Recommendation on the transport of dangerous goods, tests and criteria" [1] and was developed by Pakulak and Anderson [2, 3]. According to the UN manual, the test simulates the transport and storage situations involving external heating and provides data for classification recording to test series 1(b)iii or 2(b)iv. Also others at the Norwegian Defence Research Establishment [4] and the Materials Research Laboratory in Australia [5] have constructed an identical test facility based on the model of Pakulak and Anderson. The condition of the witness plates and the vessel are used against criteria to classify the level of severity of the explosive reaction[1].

After some preliminary SCB tests with sand and explosive substances (TNT, AN and Hexocire[2]), we noticed that some design parameters such as voltage fluctuations and thermocouples (TC) positions have a strong influence on the reproducibility of the temperature inside the SCB and therefore possibly on the test results. Therefore a programme was carried out to study the effects of parameters more carefully. Pakulak and Anderson carried out SCB experiments in a confined and unconfined set-up. To study the effects of this variation, we performed experiments in a confined and an unconfined set-up with Hexocire, TNT and AN.

The changes of the SCB test and the experimental results, together with other kinds of Cook-off tests [8, 9, 10], were presented at the "NIMIC Workshop on Cook-off" [6]. During the workshop it became obvious that besides mitigation and threats of the cook-off phenomena, the severity of cook-off is of most concern, at the moment. Therefore, the construction and development of suitable tests, predicting the severity of the explosive substance under thermal threat, is very important.

Chapter 2 gives the theory used in this report to understand the influence of voltage drifts on the temperature distribution. In Chapter 3, a description of the SCB and equipment used is given. Chapter 4 contains the results of the first tests. The discussion is given in Chapter 5. Starting with a description of the problems encountered during our first experiments, this chapter contains a possible solution for some of these problems, followed by the results of a series of new tests. The last chapter contains the conclusion.

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1 These studies have been carried out under assignment A80/KL/137

2 Hexocire contains: 94.5% RDX, 5% wax and 0.5% graphite

2 THEORY

2.1 Relation between the temperature gradient, the heating rate and the thermal conductivity of a vessel wall

The relation between the temperature gradient ∇T, the thermal conductivity λ and the heating rate of a vessel wall made of steel (heated by direct contact with a band heater) can be expressed by the energy-conservation equation:

$$\rho C \frac{\partial T}{\partial t} = \nabla \bullet (\lambda \nabla T) \tag{1}$$

In one-dimensional polar co-ordinates:

$$\rho C \frac{\partial T}{\partial t} = \lambda \left(\frac{\partial^2 T}{\partial r^2} + \frac{1}{r} \frac{\partial T}{\partial r} \right) \tag{2}$$

with the initial and boundary conditions,

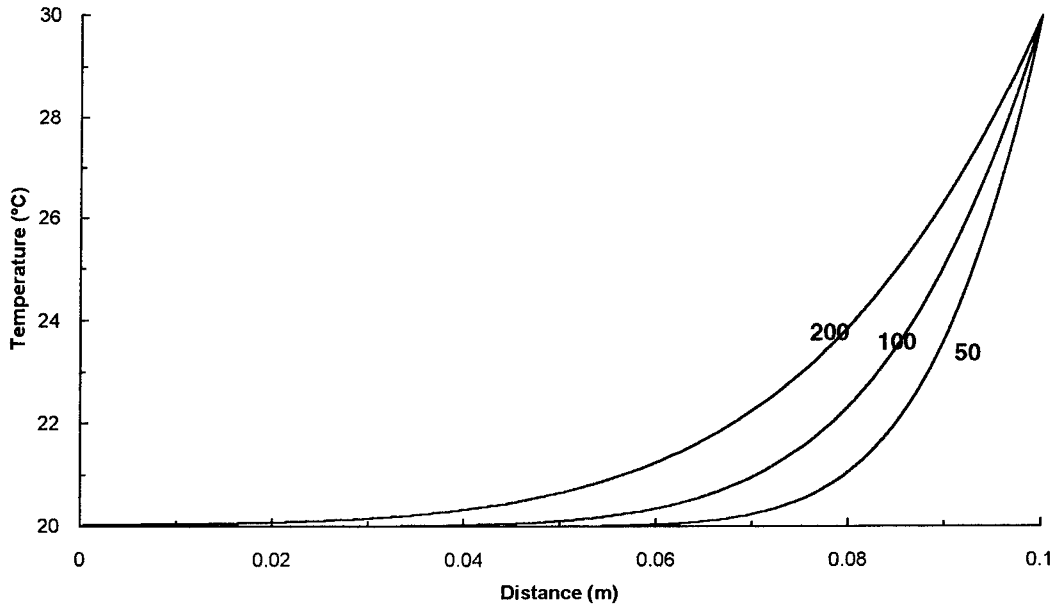
$$t = 0 : T(r, t = 0) = f(x) \tag{3}$$

$$r = R : T(r = R, t) = g(t) \tag{4}$$

where:

ρ = density [kgm^{-3}]
C = specific heat [$Jkg^{-1}K^{-1}$]
T = temperature [K]
t = time [s]
λ = thermal conductivity [$Jm^{-1}s^{-1}K^{-1}$]
and f(x) and g(t) are functions depending on the distance and time, respectively.

If the thermal conductivity λ is very large, the temperature gradient will be negligible. As a result the heat produced by the heater g(t) will be instantaneously distributed across the material. An increase of the heating rate and/or a decrease of the conductivity λ results in a higher temperature gradient near the heater. To show these effects, six one-dimensional (radial direction) computer simulations were performed on a cylinder with a radius of 0.1m. In this simulation, the initial temperature of the cylinder is 20 °C. The cylinder is heated, at the boundary r = R, with a constant heating rate of 1 °C/s or 0.333 °C/s until a temperature of 30 °C is reached. Three different thermal conductivities are used in these calculations.



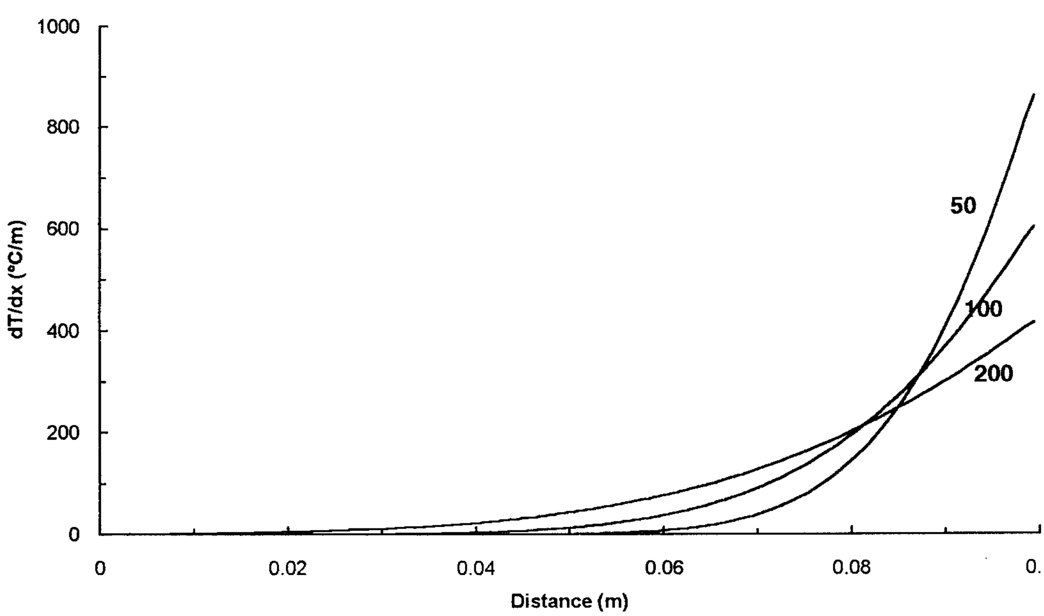
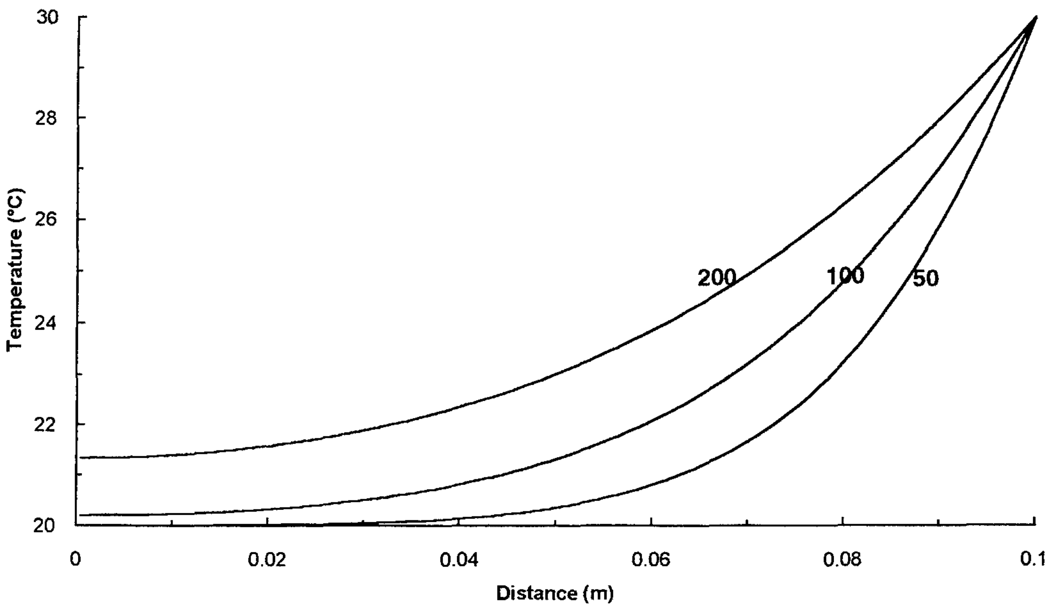


*Figure 1a and b The temperature and temperature gradient as a function of the radius of a cylinder with different thermal conductivities (λ=50, 100 and 200). Initial condition: T(r, t=0)=20 °C and boundary condition: T(r=R, t)=1*t+20 °C*

Figure 1a shows the result of three simulations at 1 °C/s with three different conductivities (50, 100 and 200 $Jm^{-1}s^{-1}K^{-1}$). In Figure 1b the temperature gradient (dT/dx) is given as a function of the radius. It shows that the largest temperature gradient is found for the smallest thermal conductivity (50) near the cylinder wall.



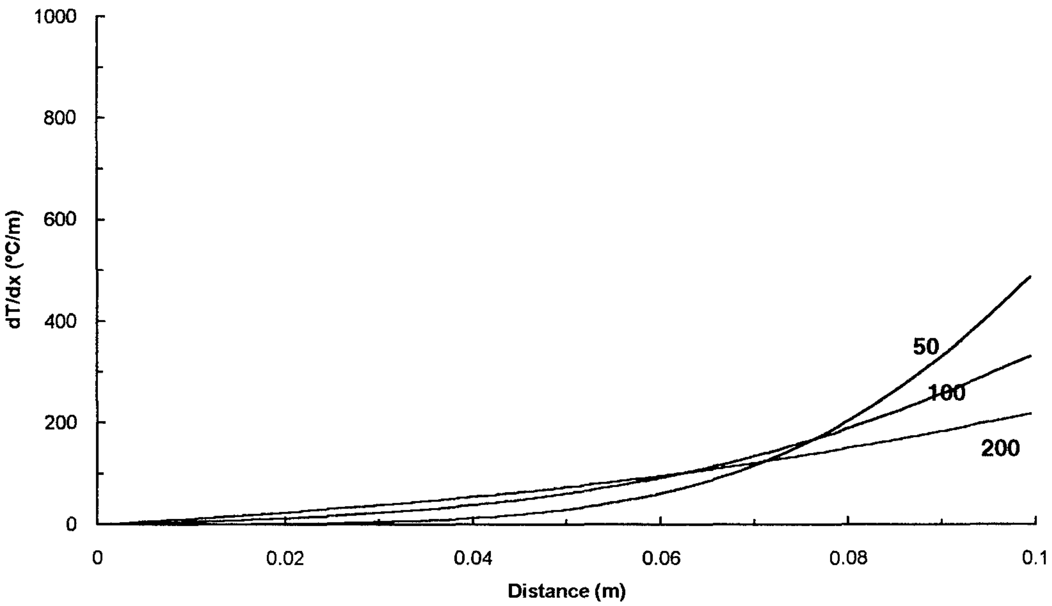


*Figure 2a and b The temperature and temperature gradient as a function of the radius of a cylinder with different thermal conductivities (λ=50, 100 and 200). Initial condition: T(r, t=0)=20°C and boundary condition: T(r=R, t)=0.333*t+20 °C*

Figure 2a and b show the temperature gradient inside the cylinder using a constant heating rate of 0.333 °C/s, and three different thermal conductivities. At the time the boundary (r=R) reaches a temperature of 30 °C, more energy has entered the cylinder compared to the results with the 1 °C/s heating rate (period of times is longer). In Figure 2b the gradients show the largest gradients for the smallest conductivity, just as in Figure 1b. Compared to Figure 1b, the gradients are smaller, which are of course due to smaller heating rates.

2.2 Voltage fluctuations and temperature

The power consumed by a band heater with a resistance R, connected to a main voltage V (VAC) is:

$$P = \frac{V^2}{R} \tag{5}$$

The temperature rise ΔT of a body with a heat capacity of C, consuming a power P for a time interval Δt is:

$$\Delta T = \frac{P\Delta t}{C} \tag{6}$$

The amplitude of the main voltage shows fluctuations with different time constants: fluctuations with time constant of a few hours in the order of ± 10 V (called ΔV) and smaller fluctuations with a time period of a few seconds and a magnitude of $\pm$ 1 ΔV (called σ_{volt}). As can be seen from equation (5) and (6), the temperature rise ΔT is quadratically dependent on V. Small variations ΔV in V will result in variations in ΔT. To investigate this relationship, consider two identical heaters connected parallel to a main voltage V and a voltage fluctuation ΔV:

$$P_{\overline{V}} = \frac{2\overline{V}^2}{\overline{R}} \quad \text{if} \quad V = \overline{V}$$

and (7a, b)

$$P_{\overline{V}+\Delta V} = \frac{2\left(\overline{V}+\Delta V\right)^2}{\overline{R}} \quad \text{if} \quad V = \overline{V} + \Delta V$$

Leading to a difference in consumed power of:

$$\begin{aligned} \Delta P &= P_{\overline{V}+\Delta V} - P_{\overline{V}} \\ &= 2\frac{\Delta V}{R}\left(2\overline{V} + \Delta V\right) \end{aligned} \tag{8}$$

Taking the effective heat capacity C_{eff} (kind of average heat capacity) as the total energy produced by the two heaters divided by the temperature difference ΔT reached after a certain time Δt, we obtain:

$$C_{eff} = \frac{P\Delta t}{\Delta T} \tag{9}$$

A voltage of V+ΔV volt compared to V volt results in a difference in the temperature difference rise dT of:

$$dT = \frac{2\Delta V}{RC_{eff}} \Delta t\left(2\overline{V} + \Delta V\right) \tag{10}$$

The error $\sigma_{\Delta T}$ made in ΔT due to a voltage fluctuation of σ_{volt} and a standard deviation in the resistance σ_{res} is (see also Annex B):

$$\sigma_{\Delta T} = \left|2C\overline{V}\right|\sigma_{volt} + \left|\frac{-C\overline{V}^2}{R}\right|\sigma_{res}$$

with (11)

$$C = \frac{2\Delta t}{RC_{eff}}$$

Using a variac in our test set-up with a mean voltage of 220 V and introducing a large fluctuation ΔV of 10 V with an error of σ_{volt} = 0.5 V, we can calculate the temperature change dT due to a voltage drift of 10 V and compare it with the experiments.

As a formula, the relative temperature change at a mean voltage V due to a voltage deviation of ΔV is the quotient between formula (10) and (6) using formula (7a) resulting in:

$$\frac{dT}{\Delta T} = \frac{2 \bullet \Delta V}{\overline{V}} * 100\% \tag{12}$$

3 THE STANDARD UN SCB TEST FACILITY

3.1 SCB test facility

The TNO-PML SCB test is almost similar to the one described in the UN manual [1] and is shown in Figure 3. The vessel is heated with two 400 J/s band heaters at 240 VAC, which means twice a constant power of 336 J/s at 220 VAC and an average heating rate of 0.8 °C/s. Usually the test is equipped with one thermocouple (TC) on the inner side of the vessel at half the height of the vessel.

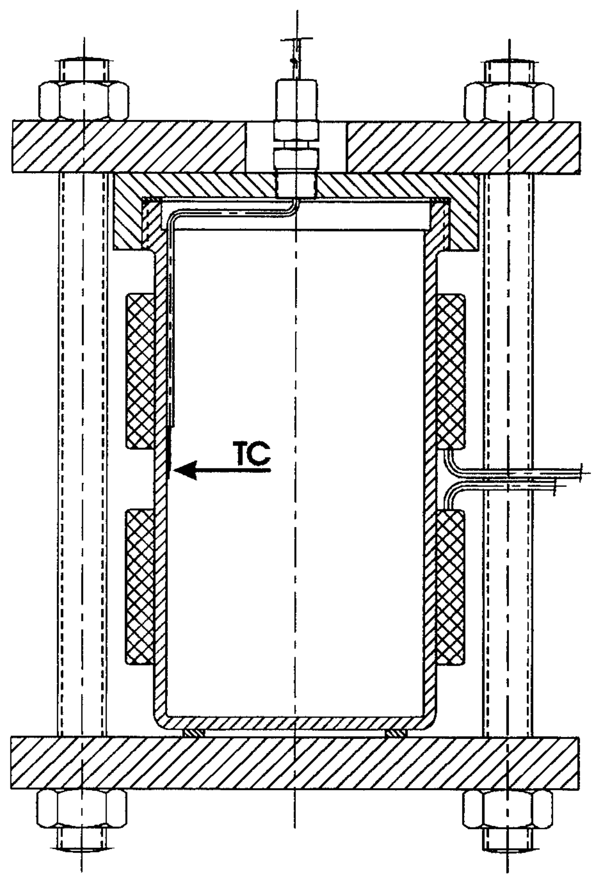


Figure 3 The TNO-PML SCB test

A few parts of the TNO-PML SCB deviate from the UN SCB test:

Part	***UN SCB***	***TNO SCB***
Top and witness plates	135 x 135 x 12.7 mm	140 x 140 x 12 mm
Thickness of the bolts	12.7 mm	12.0 mm (M12)
Thermocouple base plate	10 x 10 x 0.3 mm	9.5 x 9.5 x 0.254 mm

Other parts of the test are not precisely prescribed in the UN manual, therefore we have made the following choices:

- band heater: power 400 J/s (240 VAC), 70 mm inner diameter and a height of 38 mm;
- the TC is of chromel alumel K-type with a stainless steel coating of 0.25 mm, an outer diameter of 1.57 mm and isolated by MgO;
- the inner diameter of the fitting (feed through plug of the TC) is 1.59 mm; with this fitting it is possible to perform the SCB test in a confined configuration;
- the ring, as a sealing between the vessel and the lid, is made of Teflon;
- as a stand-off washer we prefer to use three small steel squares with a thickness of 2 mm instead of the ring to minimise the heat loss due to conduction.

A totally assembled SCB is shown in photo 1 in Annex A.

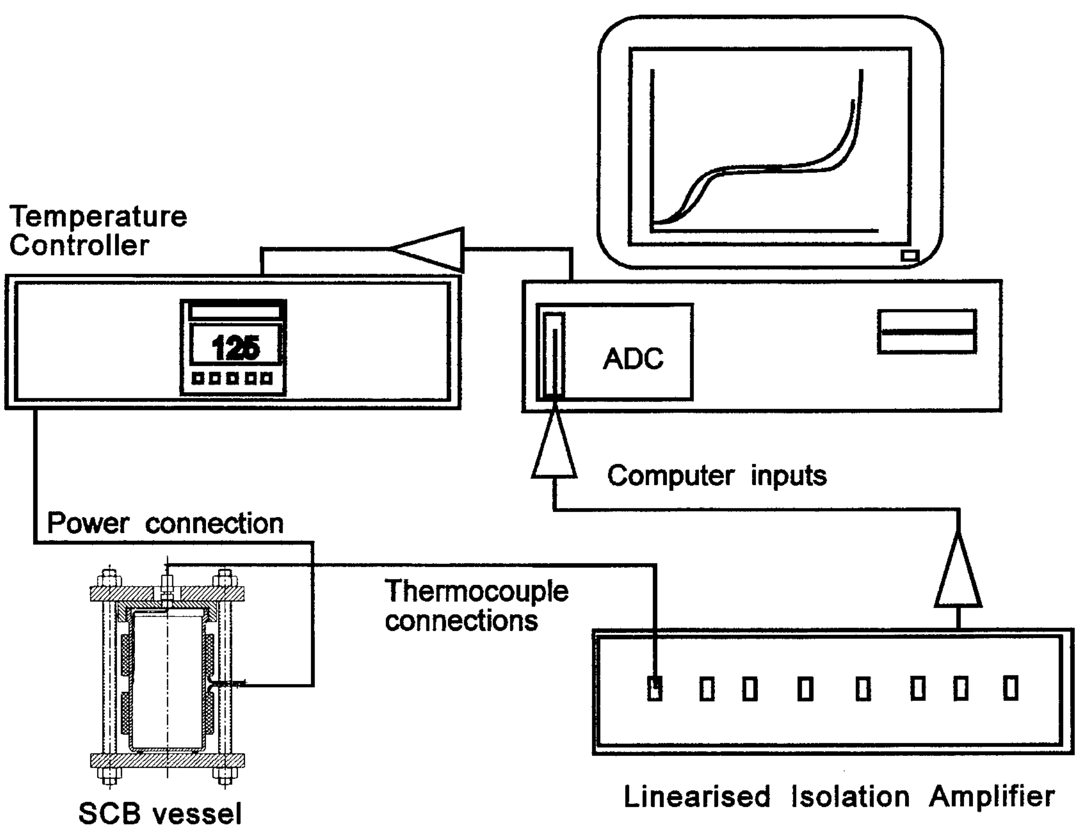


Figure 4 The total SCB test set-up

3.2 The total test set-up

Figure 4 shows the test set-up. The data acquisition is performed by a computer. In our tests we used up to eight TCs. Figure 5 shows the positions of the TC in the different used SCBs. The TC voltage is converted and linearised by Burr-Brown PCI-5B47K-05 TC linearised isolation amplifiers. Besides the signal conversion, the isolation amplifier is necessary to protect the computer and electronic system against high voltages. Subsequently the signal is led into an ADC, the digitised signal is saved on hard-disk. For data acquisition and the hardware communications, the programme "Notebook", supplied by Burr-Brown, is used. When the temperature is controlled by a PID-controller, heating rates are constant up to 1 °C/sec.

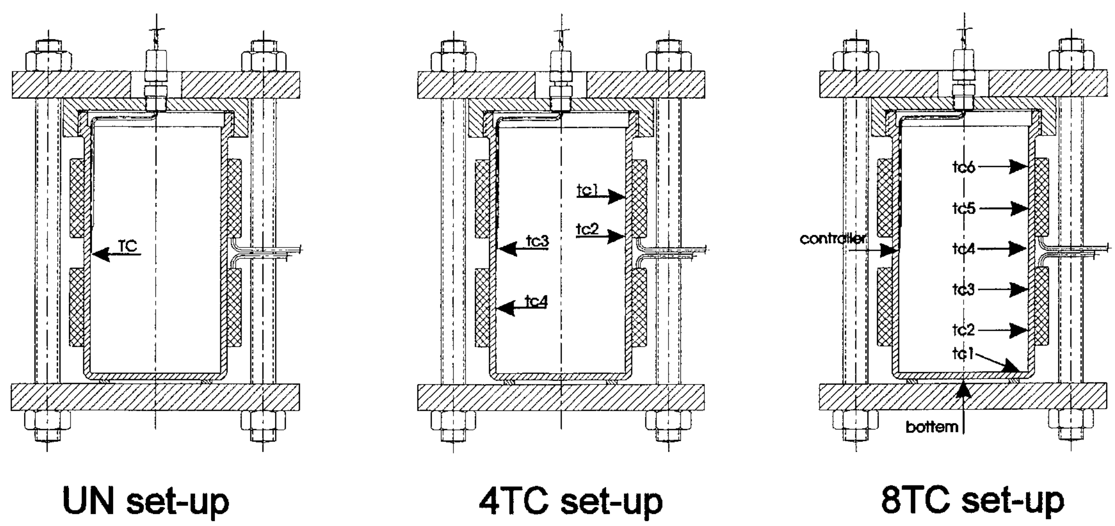


Figure 5 The thermocouple positions in different set-up configurations

4 EXPERIMENTS AND RESULTS

4.1 Preliminary tests

The set-up was tested first with no substance in the bomb. The heaters were connected to 120 and 220 VAC. Figure 6 shows the temperature-time curve for an SCB connected to a 120 VAC main voltage. After 5800 s, the TC indicates that a temperature of 225 °C can be reached. The maximum temperature of 400 °C prescribed in the UN manual is not accessible at 120 VAC.

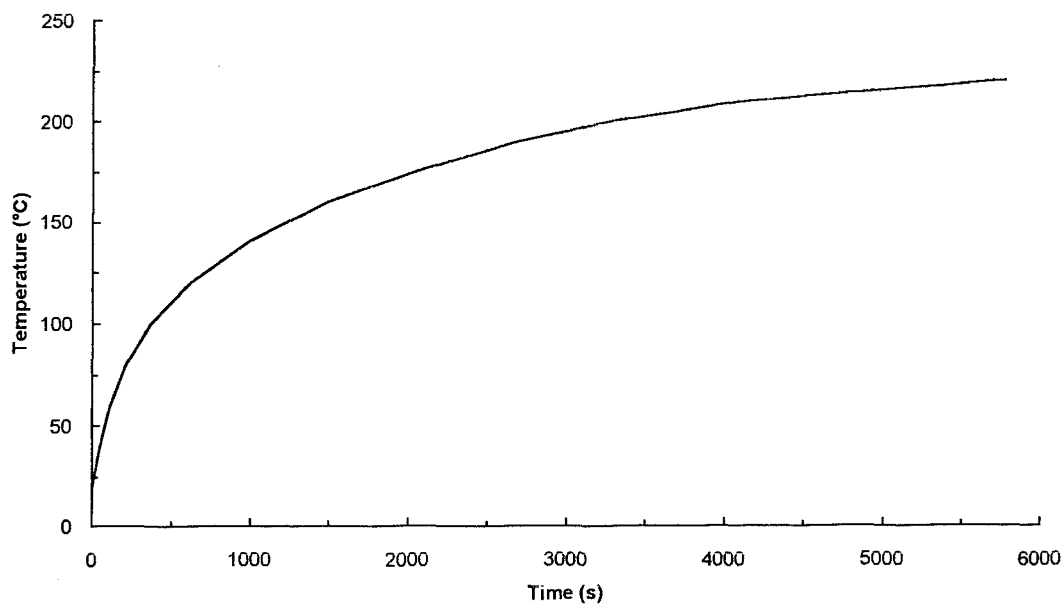


Figure 6 A temperature-time curve of an SCB (air) heated with 120 VAC

To find out the origin of this low final temperature, sources of heat loss were sought. Therefore we performed experiments at 220 VAC (instead of 120 VAC) with six different kind of washers between the witness plates and the bottom of the vessel (or lid) to measure their influence. The following washers were tested:

1. a ring of steel as described in the UN manual [1];
2. a Teflon-glass fibre ring with an outer diameter of 55 mm;
3. a frenzelite ring with an outer diameter of 55 mm;
4. three times a square plate of steel of 0.25 cm^2 and a thickness of 2 mm;
5. three times a Teflon-glass square plates with the same dimensions as number 4;
6. square plates as number 4 but this time not only at the bottom of the vessel but also between the lid of the vessel and the top-plate.

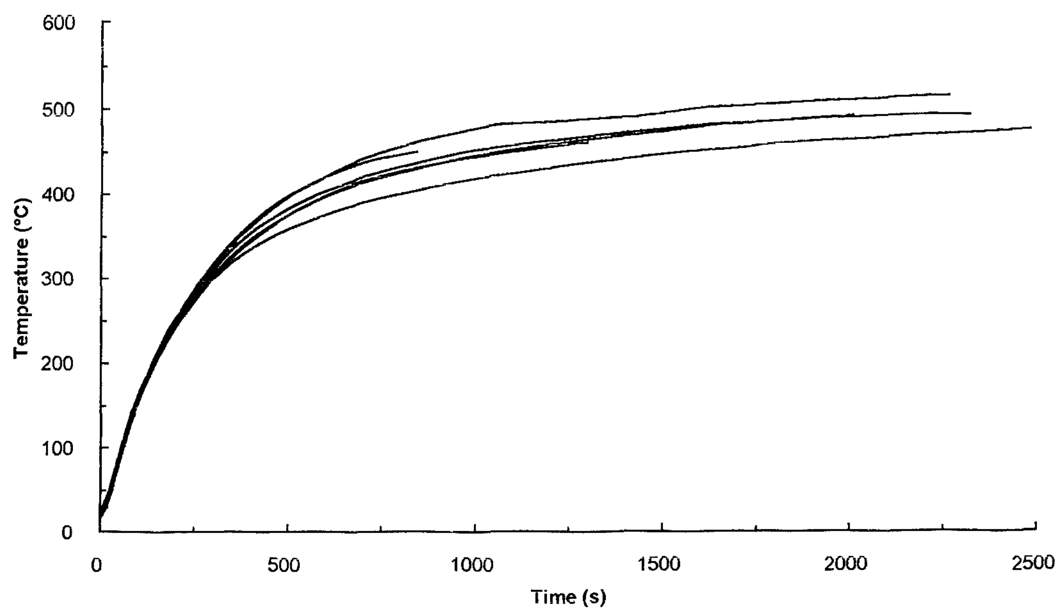


Figure 7 *The temperature-time curves of six different stand-off washers at 220 VAC*

Figure 7 shows the result of these tests. Below a temperature of 250 °C, the temperature differences are very small. Above this temperature the differences increase. The lowest maximum temperature was reached in experiment number 1 (the steel ring), the highest in the experiment using twice, three steel plates between the vessel and the witness plates. The last one could influence the test results because of the extra air gap between the top-plate and the lid. All experiments with glass fibre and frenzilite were terminated because the washers deformed during the test due to the high temperatures. The vessel came loose from the witness plates which could influence the test results. Experiment number 3 (three steel plates between the witness plate and the vessel bottom) gave the best overall result and is the closest to the original UN SCB. This solution is used in the following experiments.

4.2 Experiments with explosive substances

The following substances were used:

- TNT (flake) with a density of 1g/cm^3;
- Hexocire (94.5% RDX, 5% wax and 0.5% graphite) with a density of 1.05g/cm^3;
- AN (ANFO quality) with a density of 0.87g/cm^3.

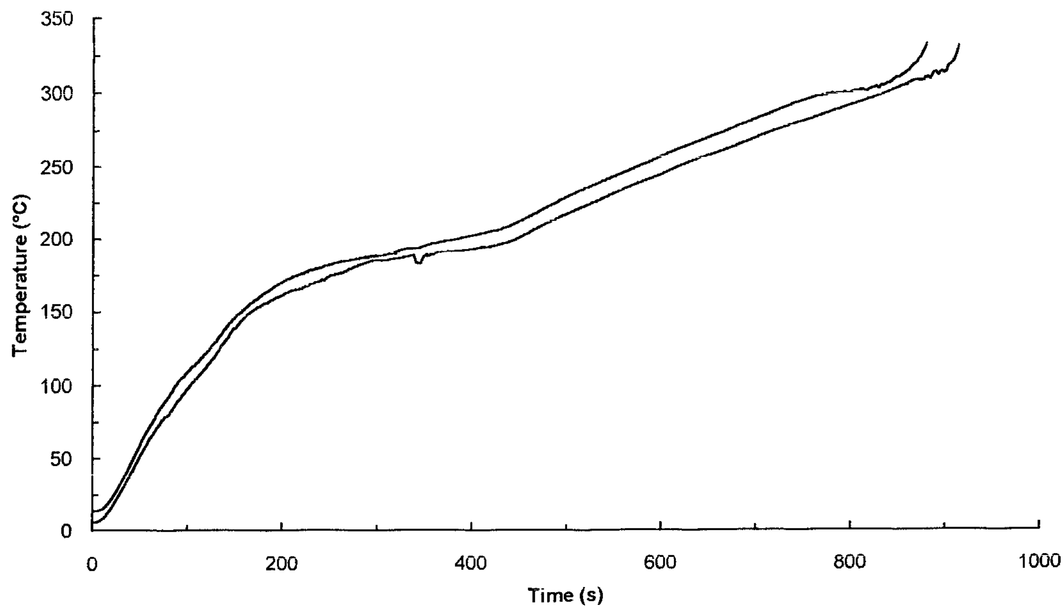


Figure 8 *The temperature-time curves of two experiments with flake TNT*

The results of the TNT experiments are shown in Figure 8. During the first 100 s of the experiment, the heating rate is almost linear. After this time a melting phase can be noticed and its influence on the temperature rise of the wall can be noticed. Most of the energy is needed for melting TNT (melting point is 80.5 °C). After 450 s, the heating rate is again almost linear. At 300 °C, slow self-heating due to exothermic decomposition of TNT is shown. The test ends with a runaway reaction followed by a severe explosion after 900 s. The small temperature difference in the two experiments is caused by a different starting temperature. The final condition of the SCB set-up is shown in photo number 2 in Annex A.

Figure 9 shows the temperature-time curve of three experiments with Hexocire. Because the melting phase of the RDX in Hexocire occurs at the same temperature as the decomposition reaction, no melting phase can be seen in these experiments. The temperature-time curve of one of the three experiments differs from the other two. Furthermore, the fragmentation of the vessel and witness plate of this experiment is more severe than of the other two. The cause can be found in a higher heating rate due to a higher main voltage, and will be discussed further in Chapter 5. Photo 3 in Annex A shows that the fragmentation of the vessel and the witness plate is more severe for Hexocire than for TNT.

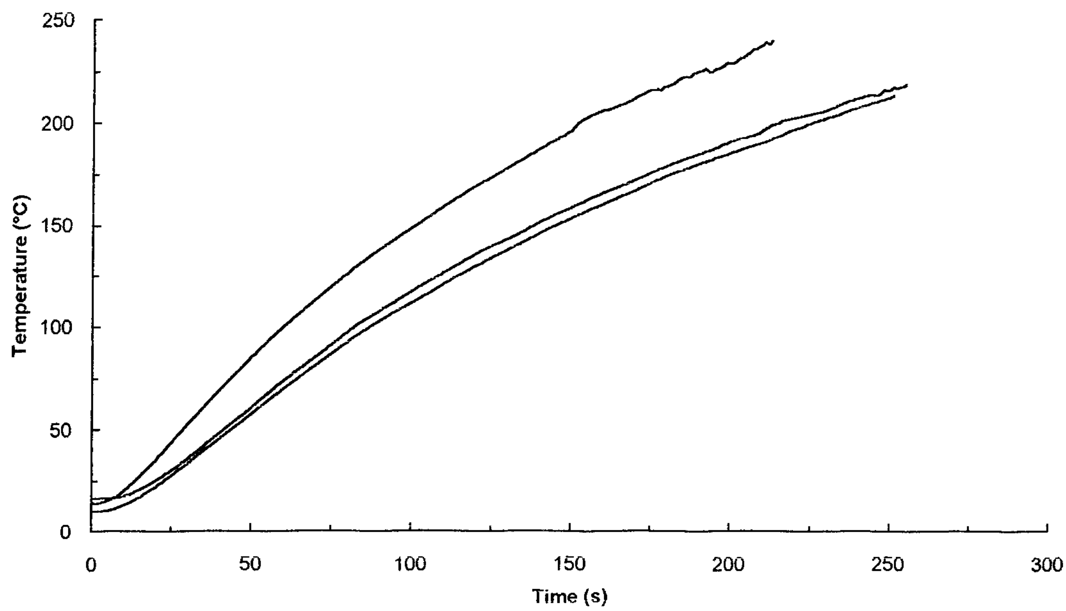


Figure 9 *The temperature-time curves of three experiments with Hexocire*

Figure 10 shows the results of three experiments with AN. The resemblance of the three experiments is quite good. There is a small deviation in the slopes during the first 150 s. This is mainly caused by a small deviation in the main voltage leading to a higher heating rate and will have its effect on the time-temperature curve in the final phase of the experiment. At 170 °C, small changes in the heating rates can be noticed. This is the start of a melting phase of AN (169.6 °C). The perturbations in the temperature of the AN1-curve starting after 300 s are probably due to gas bubbles around the TC tip. Finally, due to pressure build-up, the vessels collapsed. No damage was found to the witness plates. This is also shown in photo number 4 in Annex A.

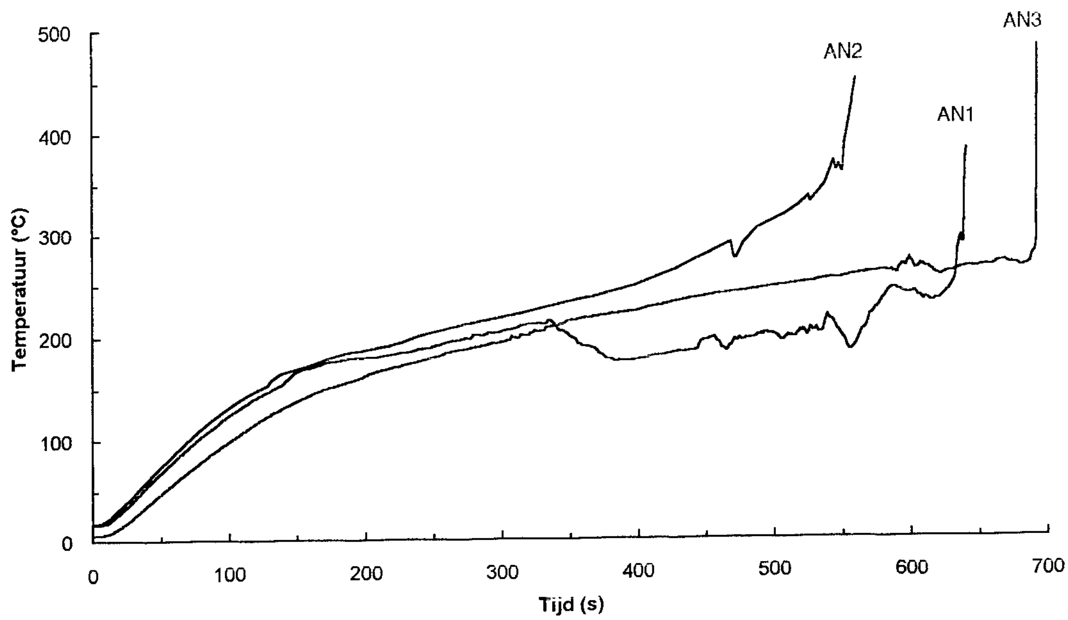


Figure 10 *The temperature time curve of three experiments with AN*

4.3 Comparison with the UN manual test results

Although comparison of our results with results given in the UN manual is difficult, they are put next to each other in Table 1. The heating rate given in the UN manual is calculated as the temperature difference divided by the time-to-explosion. No detailed information such as density or grain size was given about the used explosives in the UN manual.

Table 1 *Comparison of UN and TNO SCB test results*

	UN TN	TNO TNT 1,2
Temp. of explosion (°C)	307	332, 333
Time-to-explosion (s)	660	914, 880
Heating rate (°C/s)	0.4	0.36, 0.36
Test result (criteria)	positive	positive

	UN RDX	TNO HEX 1, 2, 3
Temp. of explosion (°C)	200	214, 240, 219
Time-to-explosion (s)	876	251, 213, 255
Heating rate (°C/s)	0.2	0.8, 1.05, 0.8
Test result (criteria)	positive	positive

Table I *Continued*

	UN AN	TNO AN 1, 2, 3
Temp. of explosion (°C)	322	290, 350, 292
Time-to-explosion (s)	780	640, 552, 692
Heating rate (°C/s)	0.4	0.4, 0.6, 0.4
Test result (criteria)	negative	positive (confined)

Comparing our test results for TNT with the criteria in the UN manual [1], we conclude that the test result is positive with a "time-to-explosion" of 15 minutes at a temperature of 330 °C. The differences in time and temperature to reaction are probably due to different heating rates. The mean heating rate in the UN manual is 0.4 °C/s, the prescribed heating rate is 0.05 °C/s (3 °C/min) and the heating rate in our experiments is 0.36 °C/s with a constant power.

The results from the Hexocire (94.5% RDX) experiments, performed at a constant power, showed a positive result, in the sense of the UN manual, after a mean time-to-explosion of about 230 s and a temperature of 220 °C. The response to the heating was a severe explosion which fragmented the vessel and the witness plate. Again differences in heating rates probably explain the differences in the time-to-explosion, though the heating rate in the experiments with UN RDX is much lower, while the explosion temperature is close to those for TNO Hexocire.

The AN test results again show differences in time-to-explosion. Using the criteria in the UN manual our result is positive in contrast to the UN results. This is probably because we performed our experiments with confined material whereas the UN tests were performed with unconfined material.

5 DISCUSSION

5.1 Introduction

It is generally known, and earlier mentioned by Hutchinson [7], that, besides the amount of confinement, the heating rate is one of the most important parameters that determines the response of a cook-off test. So a good and reproducible temperature measurement and heating rate is essential for these kinds of tests. This means that a good insight into parameters such as TC positions, heater location etc., effecting the temperature-time measurement is necessary. A closer inspection of the test data in the UN manual proves that the prescribed heating rate of 3 °C/min in the UN manual, is not used in these tests. However, the heating rate is the most varied parameter used by different laboratories:

- the UN manual prescribes a (controlled) heating rate of 3 °C/minute;
- earlier work of Pakulak [2, 3] describes an uncontrolled heating rate between 0.2 and 3 °C/s;

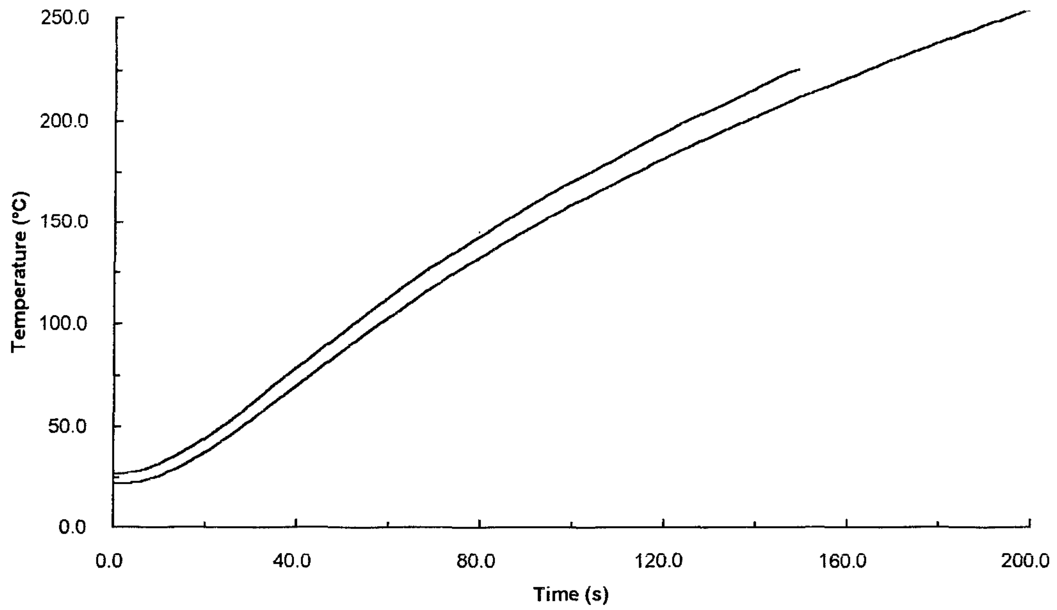
  Ingebrigtsen and Skarbøvik [4] used a controlled and almost constant heating rate of 0.49 °C/s;
- D. A. Jones and R. P. Parker [5] measured an uncontrolled heating rate of about 1 °C/s;
- TNO-PML started their first series of experiments with an uncontrolled heating rate of 0.8 °C/s.

Because of the many discrepancies we have set up a programme to study parameters influencing the temperature. In the following paragraphs we will give the results. We also show the results of some experiments with unconfined material. Next some results obtained with an improved SCB test are shown.

5.2 Reproducibility of experiments

In this paragraph we give the results of an investigation into parameters that influence the reproducibility of the temperature. The following factors are investigated:

- variation of the main Voltage;
- TC position;
- heater displacements;
- deviation of heater resistance;
- heating rate and the temperature gradients.



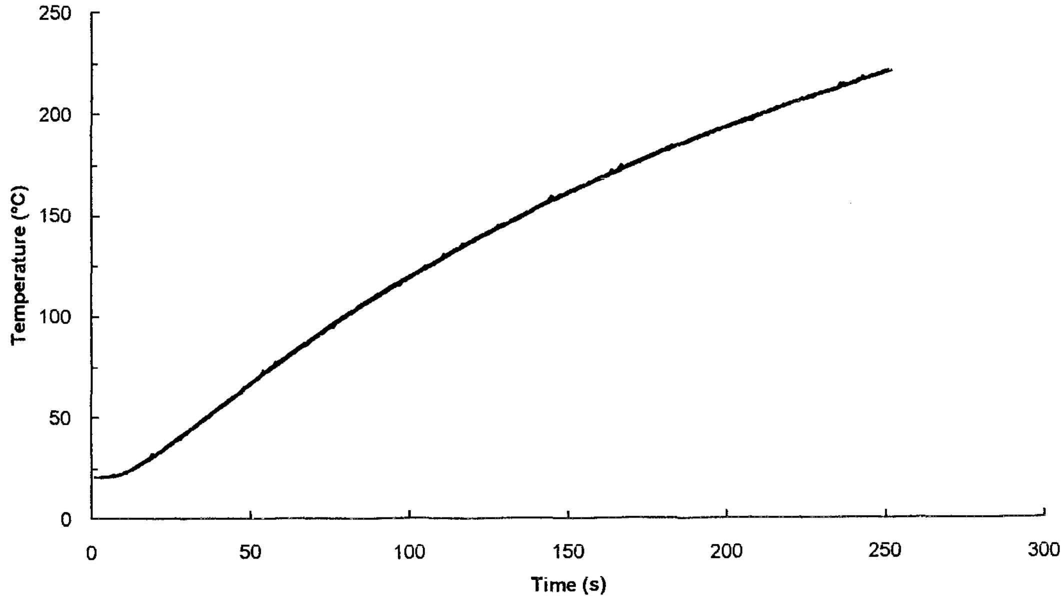


Figure 11a, b and c *Results of SCB experiments with sand connected to a main voltage of 220 VAC. Figure a with an uncontrolled heater, Figure b shows the same experiment with a controlled heater and in Figure c we see the difference in temperature of a 220 and 210 VAC controlled heater experiment*

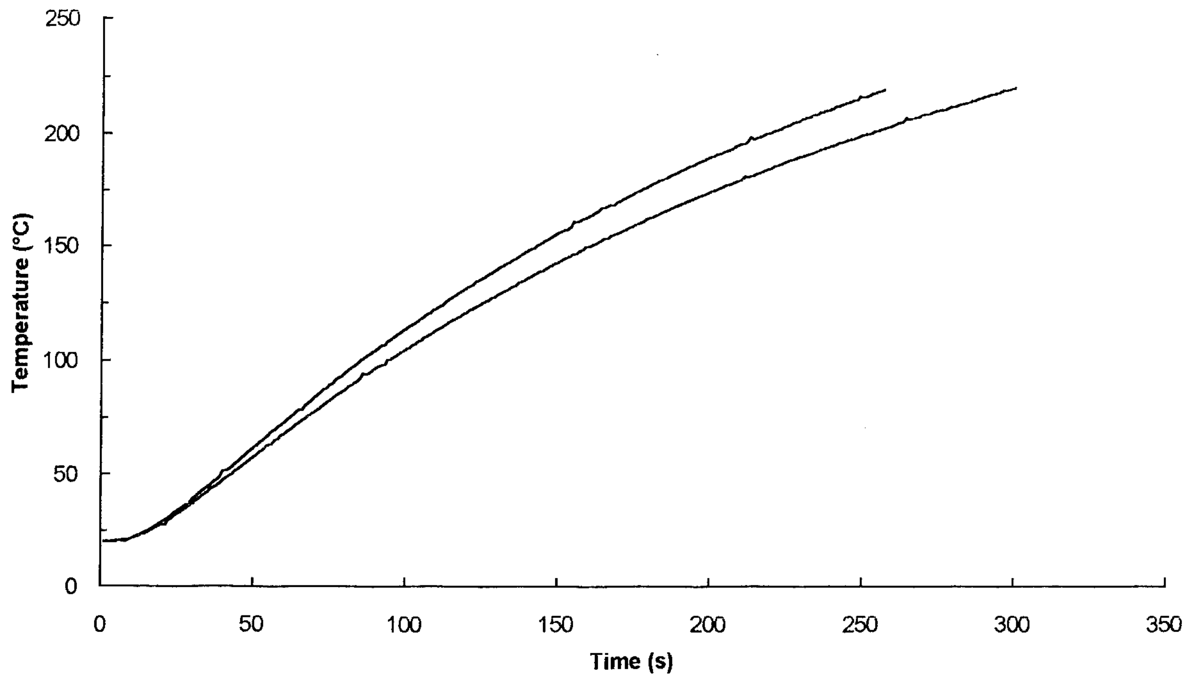


Figure 11a, b, and c *Continued*

5.2.1 Variation of the main voltage

Figure 11a shows the result of two experiments with an uncontrolled heater. Due to main voltage fluctuations (up to 20 Volts), power differences lead to a difference in temperature rise in different experiments. It is obvious that temperature differences as large as 15 °C could be noticed in the two experiments that should give similar results. Figure 11b shows the result of three temperature measurements with the heaters connected to a variac at a controlled voltage of 220 ± 0.5 VAC. The reproducibility is excellent. To show the influence of the main voltage on the time-temperature curve, two experiments were carried out (Figure 11c). In one experiment the heaters are connected to a controlled voltage of 210 VAC and in the other experiment to 220 VAC, both with a maximum variation of ± 0.5 VAC. It shows that a difference of 10 Volt results in large differences in the temperature. Comparing this result with the results in Figure 11b, the conclusion is that the heaters should be connected to a controlled voltage.

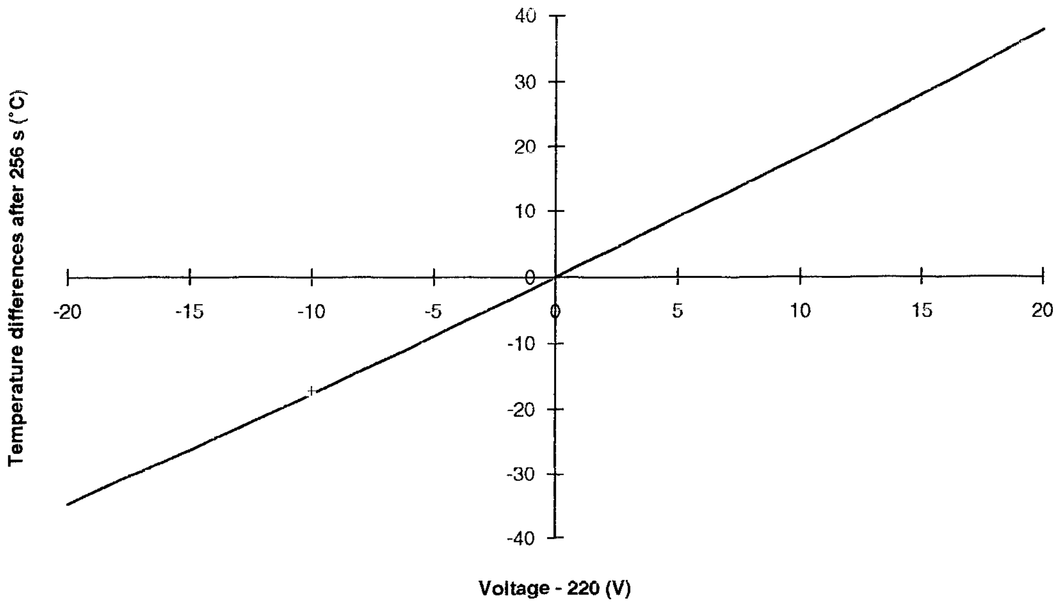


Figure 12 *The relation of the temperature difference after 256 seconds and the voltage difference (V-220) VAC. The cross gives the result of an experiment at 210 VAC*

The relation between the difference in temperature rise after a certain time, and a voltage deviation V from 220 VAC, is given by formula 10 in Chapter 2. The graphical representation of this relation is given in Figure 12, with the experimentally obtained value at 210 VAC (+). The calculated relative change in temperature with ΔV=10 V and V=220 VAC, using formula (12), is 9.1% and in agreement with the experimental value. After 256 s, at 210 VAC, the maximum calculated error between -10 and +10 VAC is 0.9 °C (σ_{volt} = 0.5 VAC, σ_{res} = 0 Ω, same SCB used twice) calculated with formula (11) (see Annex B).

5.2.2 TC position

To show the influence of the TC position, a few experiments were performed. The time-temperature curve of one experiment with a four-TC set-up is shown in Figure 13. The experiment was performed with a controlled voltage of 220 ± 0.5 VAC and TC3 in the "standard UN position" (see Figure 5). At the time TC 3 reaches a temperature of 220 °C, temperature differences up to 45 °C are measured.

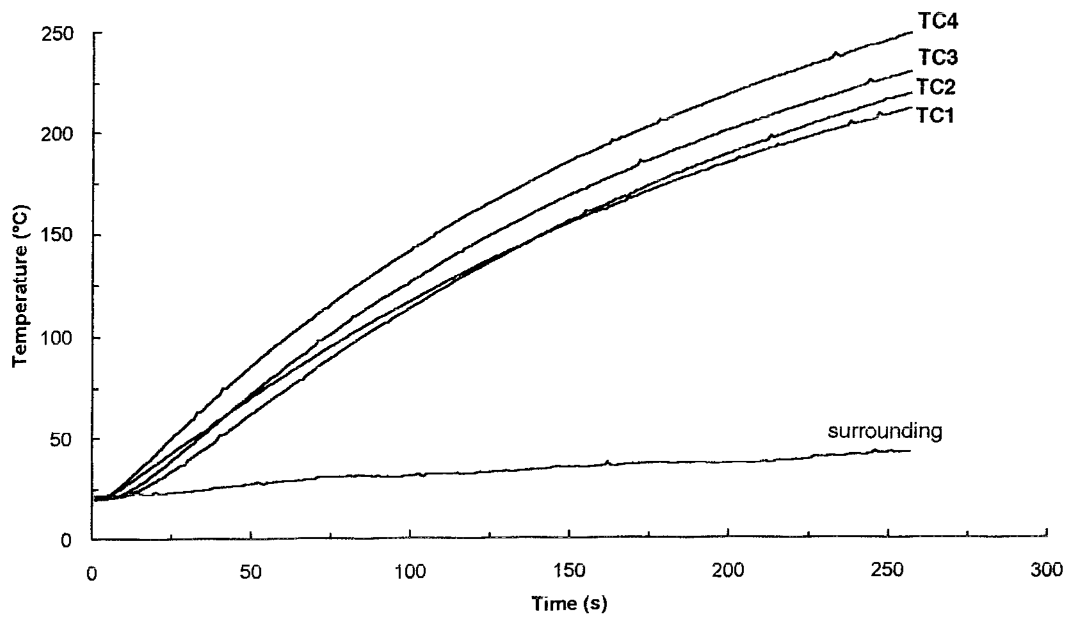


Figure 13 *Influences of thermocouple positions with TCs located at positions showed in Figure 5 b; the lower curve is the surrounding air temperature*

5.2.3 Heater displacements

In Figure 14, the effect on the temperature of a 5 mm displacement of one heater is shown (measured by a TC in the "standard UN position" (see Figure 5)). The effect of the replacement of the heater on the temperature is, of course, influenced by the positioning fluctuations of this TC.

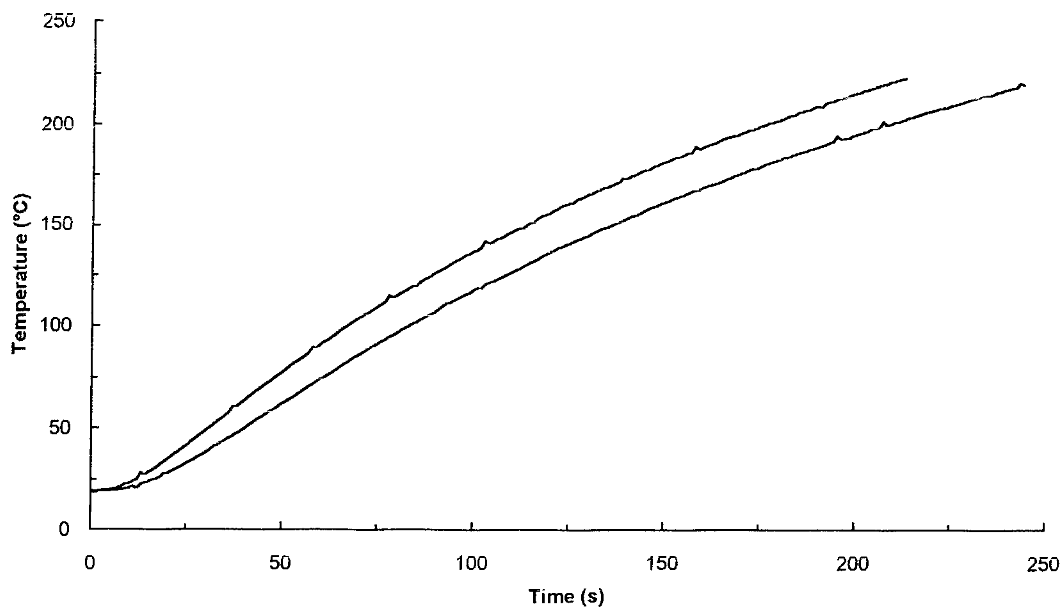


Figure 14 *Time-temperature curve differences due to a heater displacement of 5 mm*

Not only the position of the TC and the heater influence the time-temperature curve. Also, the fact that the lower part of the vessel is better insulated than the upper part will lead to a temperature gradient in the vessel.

5.2.4 Deviation in the heater resistance

The effect of a deviation in the resistance of the heaters is negligible when compared to the other effects. We measured a standard error of 1.7 Ω at an average resistance of 138.9 Ω, which results in a temperature difference of 2.5 °C after 256 seconds (calculation in Annex B).

5.2.5 Temperature gradients as a function of the heating rate

As was shown in Chapter 2 Paragraph 2.1, a relation exists between the temperature gradient, the heating rate and the thermal conductivity. To show the temperature gradient (temperature at different TC locations) at a constant heating rate and as a function of the heating rate, a series of experiments is carried out. In Figure 15, the time-temperature curves obtained with the eight-TC set-up (see Figure 5c), performed with a constant heating rate of 3 °C/minute, are shown.

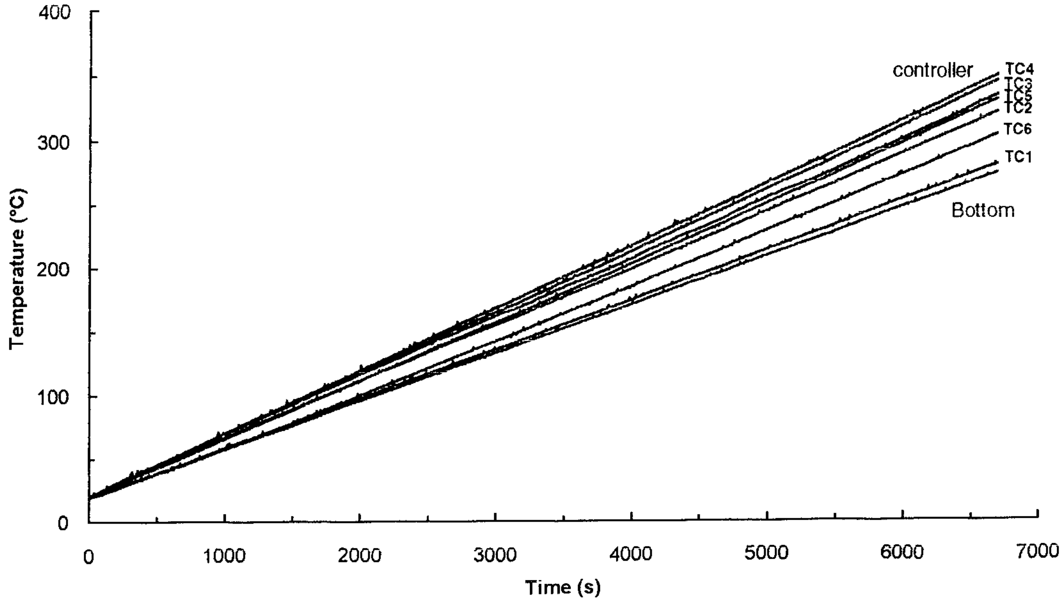


Figure 15 *The time-temperature curves of the eight-TC set-up heated with a rate of 3 °C/min*

To show that these gradients actually grow as a function of the heating rate, a series of experiments is carried out. The temperature as a function of time is controlled by the TC located at the standard UN SCB position. The absolute differences measured between this TC and the other seven TCs are given as a function of the heating rate in Figure 16. Differences up to 100 °C are measured at a rate of 0.8 °C/s (48 °C/min).

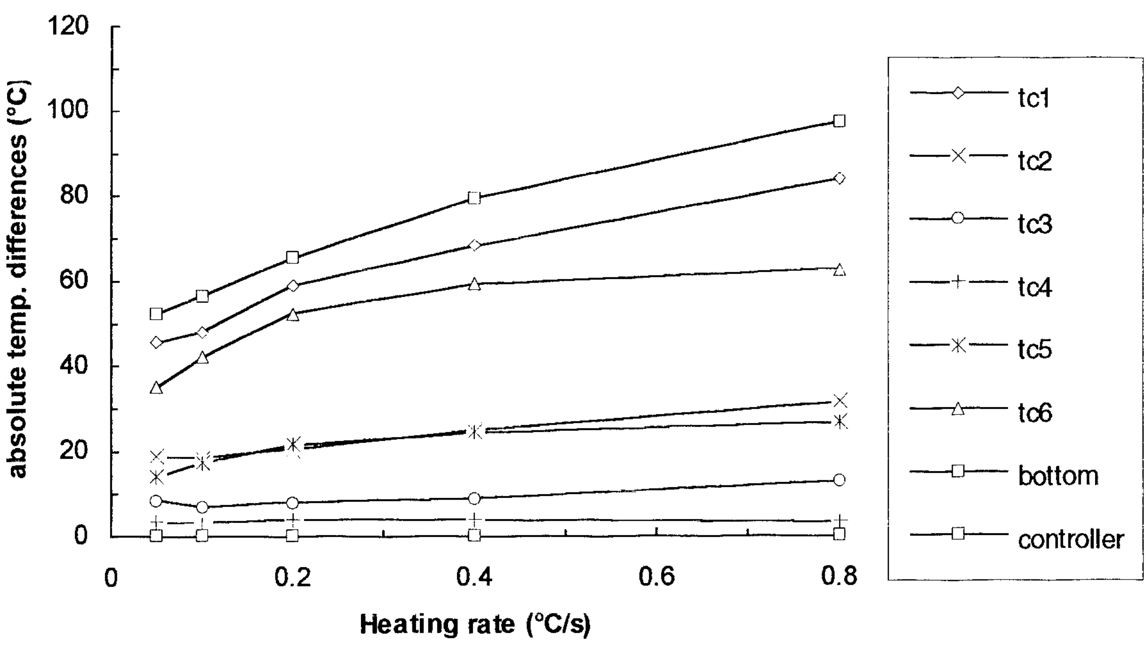


Figure 16 *Measured absolute temperature differences as a function of the heating rate of an SCB heated from 20 to 240 °C indicated by the standard UN SCB TC position*

All these effects can lead to a wrong "time-to-explosion and temperature-to-explosion" and influence the results of some critical explosive substances for classification reasons.

5.3 Experiments performed with unconfined material

5.3.1 Experiments with Hexocire

The first series of experiments with explosive substances was performed with confined material. To fulfil the UN manual SCB test description we performed two tests with explosive substances with unconfined material. These tests were carried out with a controlled voltage of 220 ± 0.5 VAC. The two experiments with RDX-wax-graphite explosive gave different results.

The first one did not explode but all Hexocire burned until the vessel was empty. A sound like a jet or torch was noticed. The SCB was fully in tact after the experiment. Using the criteria in the UN manual [1] this would give a negative test result for the RDX-wax-graphite explosive.

The result of the second experiment was different. The first part of experiment was the same, but it ended in a severe explosion. Using the criteria of the UN manual again this would lead to a positive test result for the RDX-wax-graphite explosive in contrast to the result from the first experiment.

5.3.2 Experiments with AN

An experiment with unconfined AN was not very successful. After a preheating phase the AN melted resulting in a pressure build-up inside the vessel. Thereafter the SCB started to spray all the AN through the hole into the test room, leaving a great deal of mess. After this experiment, and comparing it with earlier tests with AN, we decided not to perform any more experiments with unconfined AN.

5.4 Improvements of the SCB

5.4.1 Introduction

After these experiments we tried to improve the SCB test. Problems like voltage fluctuations, temperature gradients, reproducibility and an outside TC location in the case of pressed materials, had to be solved. In the next subparagraph the solution for the voltage fluctuations, the temperature gradient and the reproducibility will be described, followed by the description of the experiments carried out with this new test set-up. The coupling of an internal TC to a PID-controlling unit led to some controlling problems that had to be solved. With the possibility to test pressed explosives in future, we tried to find a TC location that could fulfil the reproducibility criteria. The TC is therefore located on the outside of the vessel and must produce the correct response which is important for the temperature control unit. The problems and results of these efforts and the description are given in the last two subparagraphs.

5.4.2 Changes of SCB

Several problems have to be solved: voltage fluctuations, reproducible heating rate, temperature gradients and the question whether the test has to be performed in a confined or unconfined way.

The use of a variac connected to a potentio-meter results in a constant applied voltage to the heater and minimises temperature differences due to voltage variations. A PID-controller could solve this problem as well. In this case the heating rate is maintained constant. However, maintaining a constant heating rate during a melting phase, which takes a lot of energy, sets high demands upon the heaters and the controlling unit which often results in a problematic experiment. On the other hand, using an intermediate heating rate (3 °C/min) will eliminate the expected problems with the controlling unit. Because we prefer a constant heating rate, a PID-controlling unit is used.

The temperature gradient can be minimised by using a copper shield between the vessel and the heaters. The heat of the band heaters is then distributed over a larger area of the vessel wall, because copper has a higher thermal conductivity than steel (copper 390 J/smK, steel 50 J/smK). A thicker vessel wall will partly decrease the gradients as well; temperature differences in the axial direction are smoothed due to the conductivity of steel. The drawback of this solution is an increase of the heat capacity that can be compensated for by using heaters with more power. A third possibility to reduce temperature gradients is the use of three band heaters instead of two. Because this solution is closest to the original SCB, we used three heaters coupled to the PID-controlling unit in the following experiments.

5.4.3 Experiments in new set-up

With this new test set-up we performed some experiment with new formulated PBXs. One formulation is an RDX-based PBX and the other formulation HMX based. These explosives have also been tested in a "slow cook-off test" [8] and in the "fuel fire test" [9].

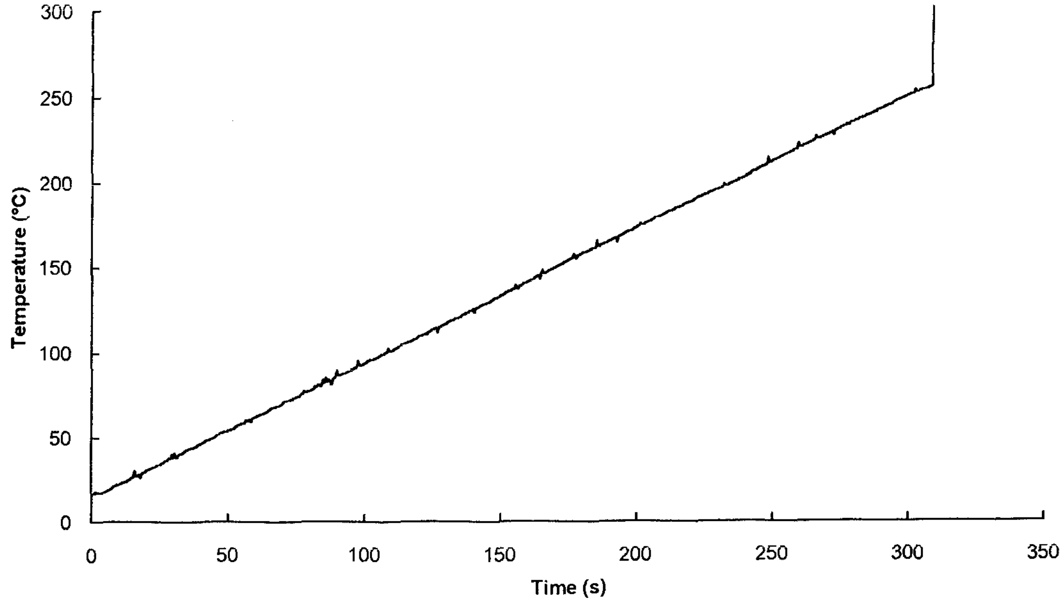


Figure 17 Temperature-time curve of an HMX-PBX filled SCB with a heating rate of 0.8 °C/s

To analyse the effects of the heating rate on the response of the explosive, the tests were performed at 0.8 °C/s and 0.05 °C/s. Using the high rate of 0.8 °C/s, no problems arose and the results were excellent. An almost linear temperature-time curve can be noticed resulting in a mild reaction after a certain time (see Figure 17). However, the lower rate of 0.05 °C/s gave unexpected problems with the control unit. Figure 18 shows the problems arising in this experiment. TC1 is the TC coupled to the PID-controller. TC2 is connected on the outside on the bottom of the SCB between the vessel and the witness plate. Up to a temperature of almost 100 °C, no problems can be seen. Just before 1400 s, problems are starting. Due to a temperature drop of the controlling TC, caused by a still unexplained factor, the controller rises the power to the heaters as can be seen by the extreme temperature rise of TC-bottom. Because of this temperature rise of the SCB and PBX, the experiment ends with an unexpected early but mild explosion.

Due to the problems arising with the control unit, to maintain a constant heating rate and the possibility to test pressed explosives in future, we decided to investigate a TC position on the outside of the SCB.

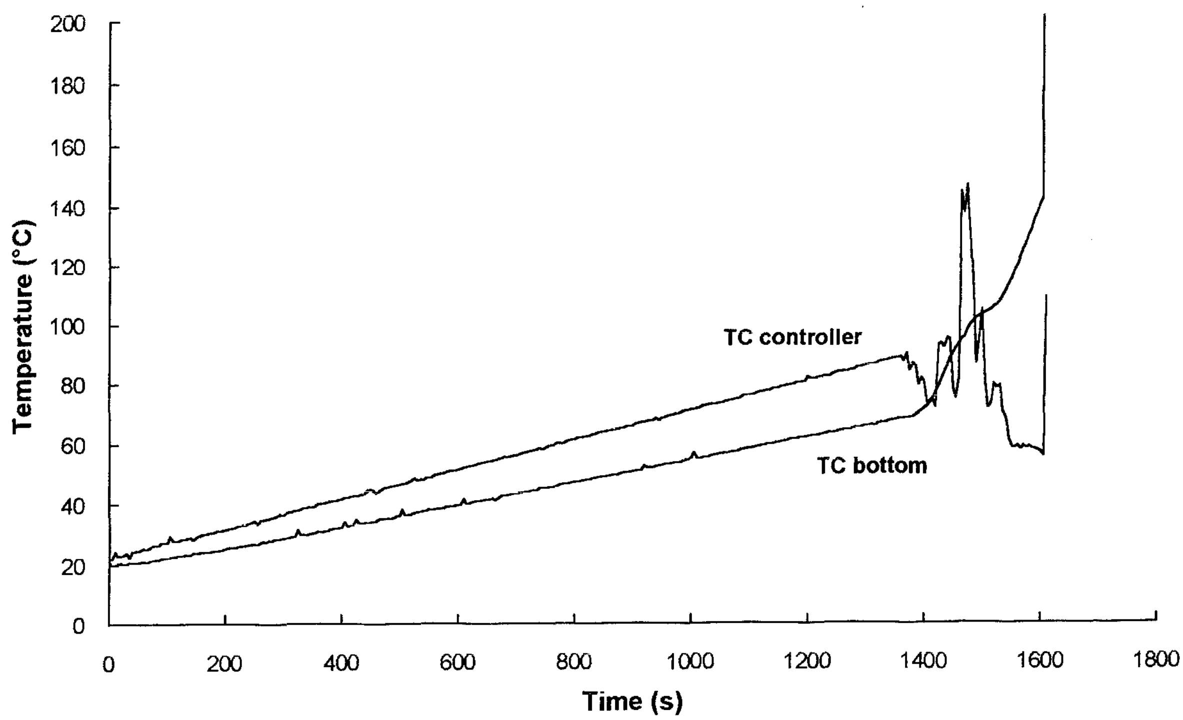


Figure 18 *Problems with an SCB test connected to a controlling unit at 0.05 °C/s*

5.4.4 TC on the outside at bottom of the vessel

In the following experiments, three heaters were used. The TC was located on the outside on the vessel on the bottom of the SCB between the vessel and the witness plate, welded to the vessel wall. The aim of the experiments was to find a relation between the temperature at the standard UN TC location (see Figure 5a) and at the bottom. Connecting the TC at the UN position to the control unit and measuring the temperature at the bottom gave the desired relation.

The problems arose in the adverse connection, the bottom TC to the controller and measuring the temperature at the UN TC location. Because of the delay in response time of the bottom TC, the controlling unit gave maximum power to the heaters. The standard TC measured an extreme heating rate, while the bottom TC did not detect any temperature rise at all. Therefore a constant and comparing heating rate, with a controlling TC at the bottom of the vessel, is not a correct solution.

5.4.5 The improved SCB, TC outside, between the connections of the middle heater

Analysing the problems arising in the last attempt, and using the theories of "Dynamical systems", we concluded that a TC should be connected close to the heat source. A solution is just between the connection of the middle heater (see Figure 19). At this location there is no direct contact with the heater. On the other hand, it is close enough to decrease the response time of the system. For reproducibility reasons, three experiments with different test set-ups using this new

TC location were performed. Figure 20 shows the results of these experiments. Because using the controller would give the same results, we performed these experiments at a constant power of 220 VAC, using the variac, measuring the temperature at the new TC location. Only small differences can be seen and therefore the new set-up seems to be very promising.

Future experiments will be performed with:

- a PID-controller at different heating rates to investigate the influences of the rate on the response of the explosive substance;
- connected to three instead of two band heaters;
- with the TC connected to the controller between the connections of the middle heater, and the explosive substance in a confined test set-up.

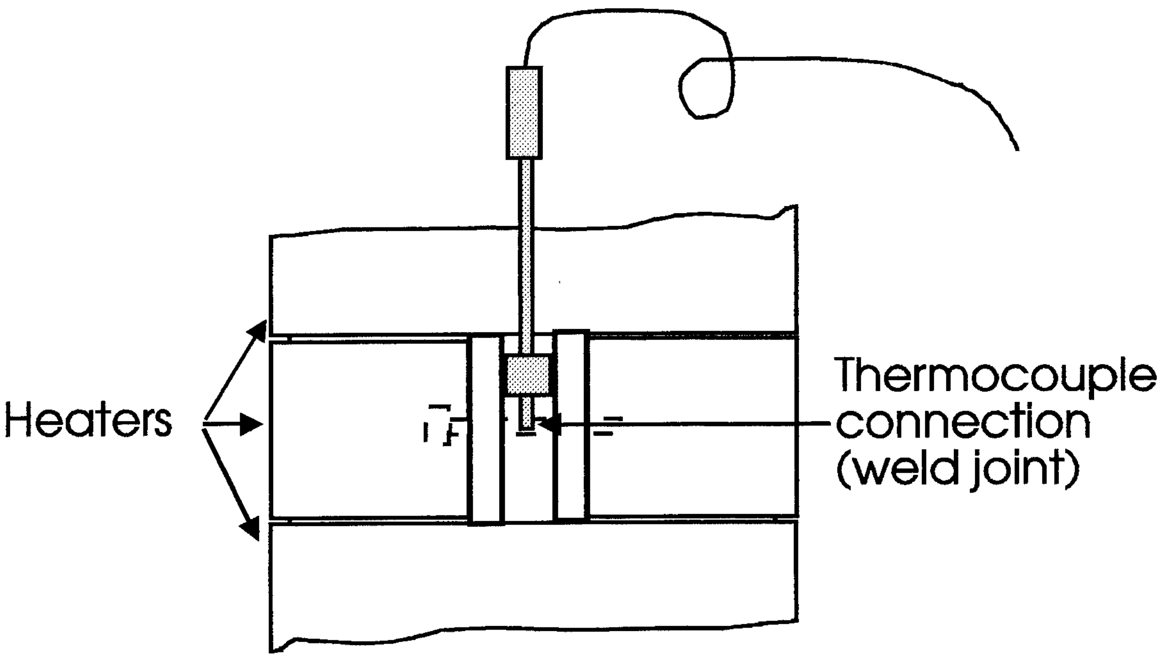


Figure 19 *TC location on the outside of the SCB, between the connections of the middle heater*

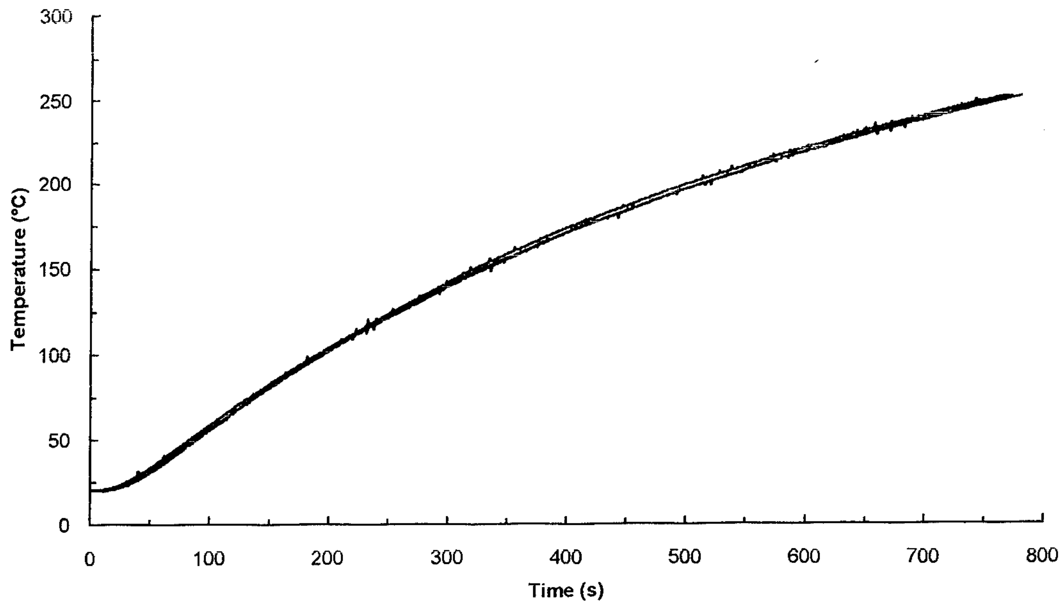


Figure 20 *Results of experiments with three different SCBs with the new TC location*

6 CONCLUSION

The Small scale Cook-off Bomb test has been established at TNO-PML. It is an easy to use test for measuring time and temperature to explosion and the severity of the explosive reaction. After some preliminary tests we noticed that some design parameters like TC and heater positions, stability of the voltage and stand-off washers have an influence on the reproducibility of the temperature measurements and therefore on the test results.

Our investigations showed that a deviation of 10 V, at a mean voltage of 220 VAC, led to a temperature change of 9%. The combination of deviations of TC and heater positions can lead to relative temperature changes in the vessel of more than 20% and are strongly influenced by the heating rate used in the test.

The tests with, several unconfined materials, turned out not to be reproducible. Therefore it was decided to perform all future tests with confined materials only.

Using the standard UN SCB test set-up, we recommend to perform the SCB test in the following set-up:

- using three instead of two band heaters to decrease the temperature gradient in the vessel wall;
- using an outside TC position between the connection-points of the middle heater (see Figure 19);
- using a constant heating rate of 3 °C/minute by using a PID-control unit. In the case of an extreme energy consumption, caused by a melting material, it is possible that even a maximum power of the heaters is not sufficient to maintain a constant heating rate. In this case a constant voltage is recommended;
- perform the test with totally confined material to obtain reproducible results.

In view of environmental concerns, the use of large-scale testing like the fuel fire test is better replaced by a small-scale test, like the SCB, as this type of testing severely reduces the pollution of air and water. In many cases, heating rates up to 1 °C/s used in these tests are possible with the SCB. Also slow rates down to 3.3 °C/hour are possible but will give an unwanted temperature gradient inside the material. For this kind of testing, cook-off set-ups like the TNO-PML Cook-off test [10] are better suited. Performing tests with intermediate heating rates gives no problem for the SCB set-up.

As mentioned earlier, besides mitigation and defining the threats of the cook-off phenomena, the severity of the response of an explosive substance subjected to a thermal threat, is important. An explosive substance subjected to a thermal threat, giving a detonation as a response at 250 °C, is much more hazardous than an explosive substance giving a mild burning at 150 °C. Therefore, the construction and development of suitable tests, predicting the severity of an explosive substance under these circumstances, is essential for the investigation of the cook-off phenomena.

7 AUTHENTICATION

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(Author)

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(Author/Research Co-ordinator)

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ANNEX A PHOTOGRAPHS

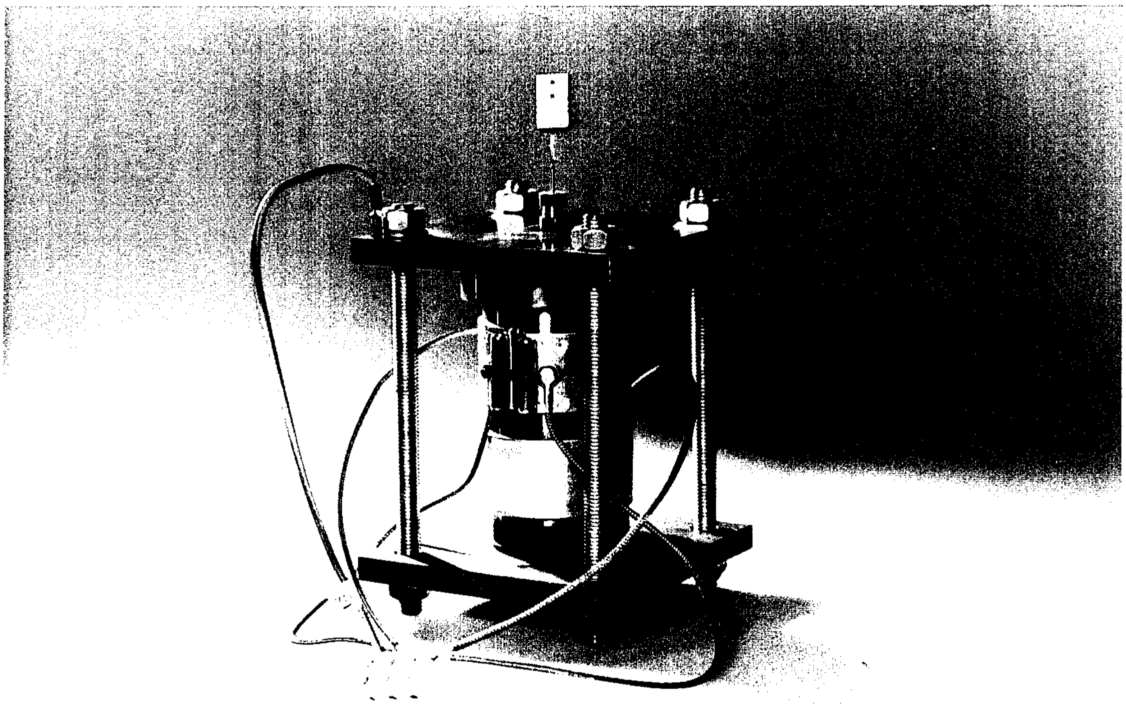

Photo 1 *The assembled SCB test object before an experiment*

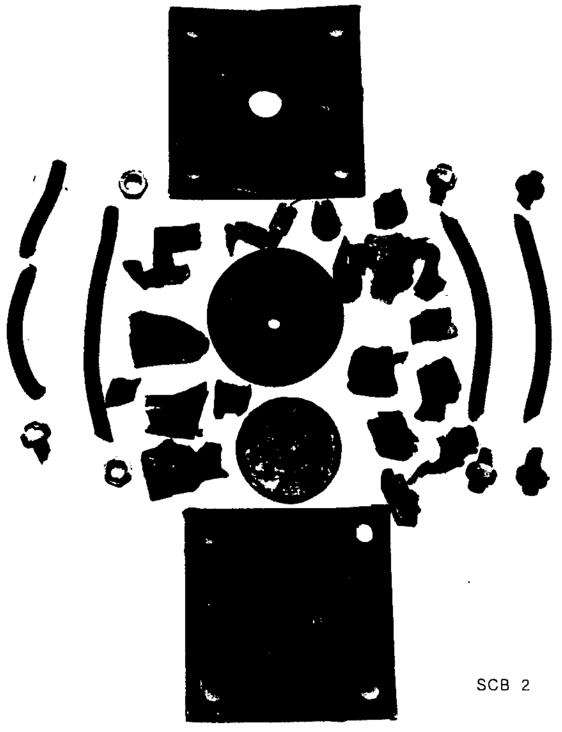

Photo 2 *The result of an SCB experiment with flake TNT (confined)*

Photo 3 *The result of an SCB experiment with Hexocire (confined)*

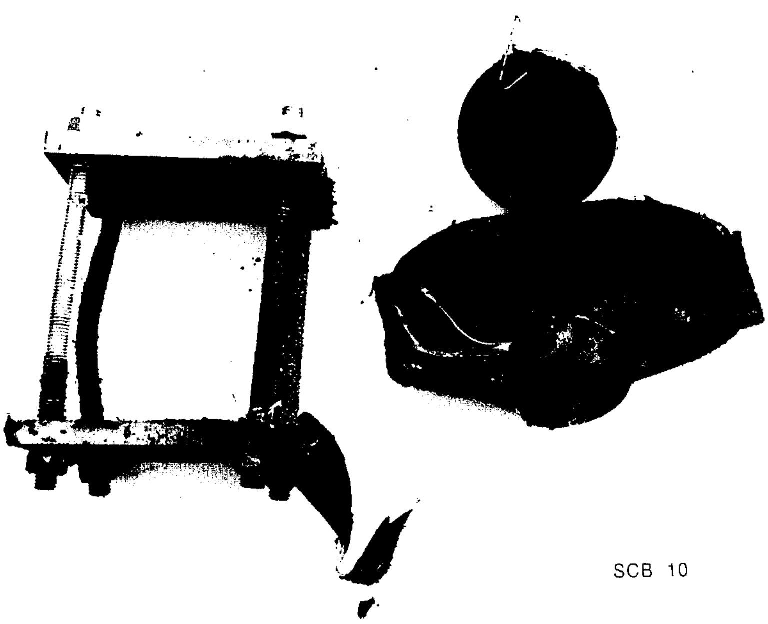

Photo 4 *The result of one SCB experiment with AN (confined)*

ANNEX B CALCULATIONS OF THE ERRORS OF TEMPERATURE DIFFERENCES DUE TO THE DEVIATION IN RESISTANCE OF THE HEATERS AND FLUCTUATIONS OF THE MAIN VOLTAGE

Taking the formula for the temperature as a function of the voltage V and resistance R (with two heaters):

$$P = \frac{V^2}{R} \bullet 2$$

and

$$T - T_0 = \frac{P\Delta t}{C_{eff}}$$

gives

$$T - T_0 = 2 \bullet \frac{V^2 \Delta t}{RC_{eff}}$$

The error in ΔT as a result of the error in the resistance and the error in the main voltage is:

$$\sigma_{\Delta t} = \left|\frac{\partial \Delta T}{\partial V}\right| * \sigma_{volt} + \left|\frac{\partial \Delta T}{\partial R}\right| * \sigma_{res} \tag{13}$$

After some mathematics this lead to a formula like formula 11:

$$\sigma_{\Delta T} = \left|2C\overline{V}\right|\sigma_{volt} + \left|\frac{-C\overline{V}^2}{R}\right|\sigma_{res}$$

with (11)

$$C = \frac{2\Delta t}{RC_{eff}}$$

In our experiments the remaining values are:

t	= 256 s
R	= 139.8
C_{eff}	= 897.42 $Jkg^{-1}K^{-1}$
$\overline{V}$	= 220VAC
ΔV	= -10 VAC

Resulting in an error in T of:

$$\begin{aligned} &\sigma_{\Delta T} = |1.8| \bullet \sigma_{volt} + |1.43| \bullet \sigma_{res} \\ &\sigma_{\Delta T}(\sigma_{volt} = 0.5V, \sigma_{res} = 0.0\Omega) = 0.9°C \\ &\sigma_{\Delta T}(\sigma_{volt} = 0.0V, \sigma_{res} = 1.72\Omega) = 2.5°C \end{aligned} \tag{14}$$

Considering experiments with different SCBs, the error in heater resistance is σ_{res}=1.72 Ω and the error of the checked voltage is about σ_{volt}=0.5 VAC. In measurements with the same SCB, the error in the resistance is negligible and the error in the main voltage remains 0.5V.

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15. ABSTRACT (MAXIMUM 200 WORDS (1044 BYTE))

The construction of the Small-scale Cook-off Bomb (SCB) test facility of test series 1 and 2 of the UN manual [1] was started at TNO Prins Maurits Laboratory during the spring of 1991. After a series of tests with TNT, RDX and AN, it was noticed that design parameters such as voltage deviation, stand-off washers, thermocouple and heater positions could have a strong influence on the temperature measurement and therefore on the test results. An investigation into these parameters was carried out. The combination of these influences can lead to a relative deviation of the real temperature of 30 %. Recommendations are given and tested to change the SCB test resulting in a better reproducibility and more reliable test.

16. DESCRIPTORS	IDENTIFIERS
Tests Time Measurement Explosions Explosives	Cook-off Bomb Temperature Measurement

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